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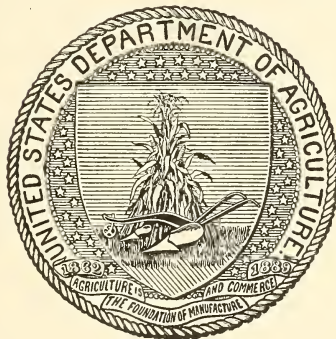
BUREAU OF SOILS—BULLETIN NO. 24.

MILTON WHITNEY, Chief.

THE CENTRIFUGAL METHOD OF MECHANICAL SOIL ANALYSIS.

BY

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LETTER OF TRANSMITTAL.

U. S. DEPARTMENT OF AGRICULTURE,
BUREAU OF SOILS,
Washington, D. C., August 31, 1904.

SIR: I respectfully submit herewith the manuscript of a paper entitled "The Centrifugal Method of Mechanical Soil Analysis," which has been prepared in response to numerous requests for a description of the method of mechanical soil analysis used by the Bureau, our earlier publications on this subject being out of print.

In addition to a description of the method of analysis the paper contains an account of certain investigations of various features of the method, regarding which there has been some uncertainty. For the information of students and others interested in the subject of mechanical analysis, a brief description of other methods at present used in the United States is appended. An account is also given of the chromic acid digestion method for determining organic matter in soils, as used by this Bureau at the present time.

Respectfully,

MILTON WHITNEY,
Chief of Bureau.

Hon. JAMES WILSON,
Secretary of Agriculture.

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THE CENTRIFUGAL METHOD OF MECHANICAL SOIL ANALYSIS.

INTRODUCTION

The mechanical analysis of a soil consists (1) in the separation of the soil particles into groups dependent upon the size of the grains, and (2) in the determination of the percentage by weight of the particles constituting each group. The limits of these groups are arbitrarily chosen, with reference (*a*) to the ease in making the separation, and (*b*) to the importance of the particles of any given size in determining the physical characteristics of the soil.

Virtually all the methods of mechanical analysis proposed take advantage of the different rates of subsidence of particles of different diameters when suspended in water. In the Osborne^{*a*} and the Knop^{*b*} methods the particles are allowed to subside under the influence of gravity in stationary columns of water. In the Schöne^{*c*} method and the Hilgard^{*d*} elutriator method the subsidence of particles of a given diameter, under the influence of gravity, is compensated by the upward flow of a column of water, which carries off particles of smaller diameter. The centrifugal method,^{*e*} as described by Snyder,^{*f*} Hopkins,^{*g*} and this Bureau,^{*h*} is essentially a sedimentation method similar to that employed by Osborne, except that centrifugal force is used to hasten the subsidence. In the centrifugal elutriator of Yoder^{*i*} the principle employed is the same as in Hilgard's elutriator method, centrifugal force being used instead of the force of gravity to effect the subsidence of the particles.^{*j*}

^{*a*} Ann. Rep. Conn. Expt. Sta., 1886, p. 141; 1887, p. 144; 1888, p. 154.

^{*b*} Wiley's Agricultural Analysis, vol. 1, p. 189.

^{*c*} Bulletin de la Société des Naturalistes de Moscou, 40, p. 324. Wiley's Agricultural Analysis, Vol. I, p. 212.

^{*d*} Ann. Rep. Cal. Expt. Sta., 1891-92, p. 243.

^{*e*} So far as we know, centrifugal force was first made use of in connection with mechanical soil analysis by Professor Whitney, in 1892, at Clifton, Md. He used centrifugal force to hasten the process of sedimentation in the Osborne beaker method, using a Babcock hand separator for this purpose.

^{*f*} Ann. Rep. Minn. Expt. Sta., 1895, p. 62.

^{*g*} Proc. Asso. Off. Agrl. Chemists, Bulletin 56, Division of Chemistry, U. S. Dept. of Agr., 1898, p. 67.

^{*h*} Field Operations of Div. of Soils, 1899, Report No. 64, U. S. Dept. of Agr., 1900, p. 173; Yearbook, U. S. Dept. of Agr., 1900, p. 397.

^{*i*} Bulletin 89, Utah Expt. Sta., 1904.

^{*j*} A very complete account of the foreign methods of mechanical soil analysis may be found in Wiley's Principles of Agricultural Analysis, Vol. I.

King^a has described a method of determining what he terms the "effective diameter" of the soil grains in a given soil, based upon the rate of flow of air through the dry soil. This determination would not properly be called a mechanical analysis in terms of the definition given above. It leads, however, to a classification of soils based upon the size of the soil grains, and possesses the distinct advantage of expressing the texture of a soil by a single numerical quantity. Another method of accomplishing this result, based upon the ordinary mechanical analysis of the soil, will be given in a subsequent paper.

This bulletin contains a description of the centrifugal method of mechanical soil analysis as used in this Bureau, together with the results of an investigation of the possible sources of error. The procedure followed by the Bureau in collecting and preparing the samples for analysis is stated, and a brief description is given of other methods of mechanical analysis, several of which have been extensively used in the United States. The chromic-acid method, now employed by the Bureau for determining organic matter, is also described.

THE CENTRIFUGAL METHOD OF MECHANICAL SOIL ANALYSIS.

COLLECTION AND STORAGE OF SAMPLES.

Great care must be observed in the collection of soil samples, in order that they may be characteristic of the area or the conditions which they represent. The heterogeneity of nearly all soils renders a sample taken at random practically worthless. The area under examination should first be thoroughly explored, using an auger or sampling tube for this purpose, after which samples should be taken which are typical of the conditions in various parts of the area.

In the soil survey the samples are taken with the aid of a 1½-inch wood auger, the shank of which has been lengthened to 3 feet. The sample is divided into soil and subsoil, in case any distinction between the two is apparent. No arbitrary division is made between the soil and subsoil, as far as depth is concerned. The sample is usually taken to a depth of 3 feet, and is divided into as many portions as there are changes in texture, the depth and thickness of each stratum being noted.

Composite samples are not used in the present work of the Bureau, except in so far as it may require several borings within a few feet of each other to secure a sample of suitable size. A sample should consist of at least 1 pound of material, and should either be taken when the soil is rather dry, or else be broken into small lumps and air dried before shipment. Otherwise heavy samples will dry into hard masses that are very difficult to prepare for analysis. The Bureau samples are shipped in cloth sacks, accompanied by suitable identification

^a Ann. Rep. Wis. Expt. Sta., 1897, p. 123.

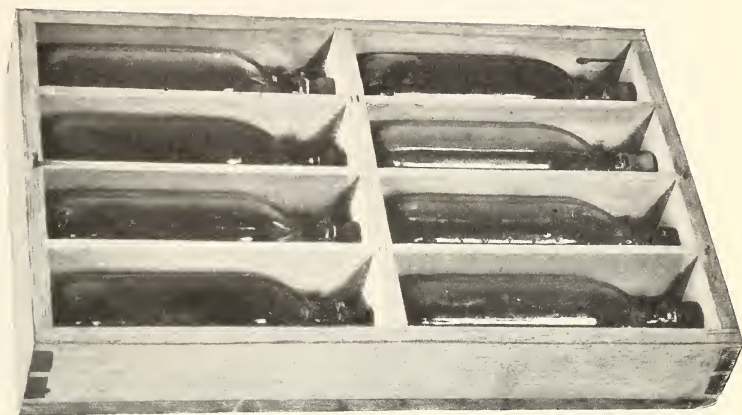


FIG. 1.—SHAKER TRAY, CONTAINING BOTTLES.

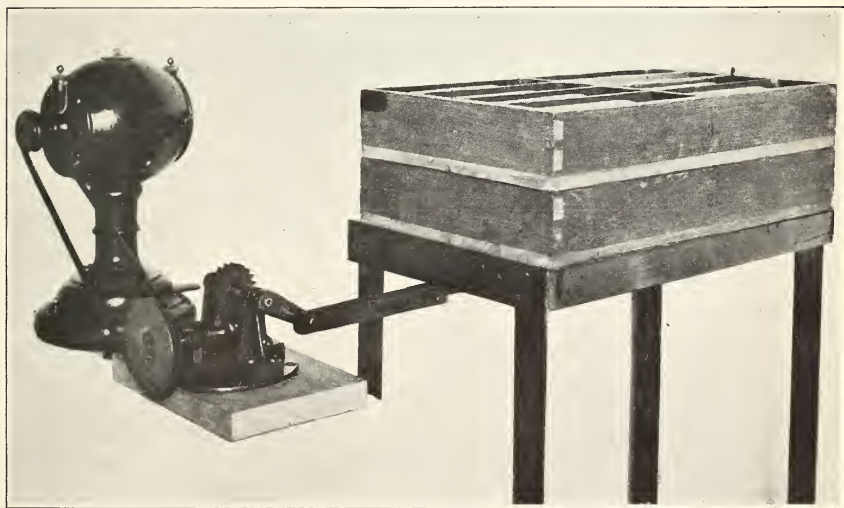


FIG. 2.—MECHANICAL SHAKER USED IN PREPARING SOILS FOR MECHANICAL ANALYSIS.

tags. They are stored in pint fruit jars, with tight-fitting tops, and are given serial numbers, by which they can subsequently be easily identified with the aid of a card catalogue.

PREPARATION OF THE SAMPLE FOR MECHANICAL ANALYSIS.

It has been the practice of this Bureau to make a mechanical analysis of that portion of a sample which passes through a 2 mm. sieve, to which the term "fine earth" has been applied. Many of the samples reach the laboratory in a lumpy condition, so that it is necessary to break up the sample by mechanical means before it can be passed through the sieve. A heavy wooden rolling-pin, 4 inches in diameter, is used for this purpose. The use of a mill or pestle is not to be recommended, as the larger grains are apt to be crushed by such a process. In case the soil contains any appreciable amount of gravel which will not pass a 2 mm. sieve, it is weighed and reported in terms of per cent by weight of the whole sample. The mechanical analysis is confined to the portion passing through the sieve, and the amounts of the various groups found are expressed as percentages of the fine earth, without reference to the coarser material which may be present.

In subsampling for mechanical analysis the portion of the sample passing the 2 mm. sieve is spread out upon a piece of heavy paper and thoroughly mixed with a large spatula. The sample is then divided into four parts, and one portion is again thoroughly mixed, from which a sample of from 10 to 20 grams is removed for mechanical analysis. This sample is dried at 110° C. and from the dry material a 5-gram portion is weighed out for analysis.

DISINTEGRATION OF THE SOIL BY THE MECHANICAL SHAKER.

Before making an analysis it is necessary to disintegrate any aggregations of particles which exist in the dry soil. This is best accomplished by shaking the samples with water for a few hours. The 5-gram samples are introduced into 250 cc. cylindrical round-bottomed sterilizing bottles, each containing approximately 75 cc. of distilled water. Ten drops of strong ammonia (sp. gr.=0.9) are added to each bottle, which is then closed with a well-fitting rubber stopper. The bottles are placed in individual compartments in trays, each tray holding eight bottles (see Pl. I, fig. 1), which corresponds to the number of samples usually analyzed at a time.

The shaker illustrated in Plate I, figure 2, consists of a platform for carrying the trays, resting upon four flexible wooden supports, to which a reciprocating motion is imparted by a crank. Each tray is provided with a pin at either end which engages with the tray above it, so that six trays may be placed upon the shaker at one time, all easily accessible. The shaker is driven by a one-sixteenth horse-power motor which is belted to a fiber worm reducing gear, provided

with a crank to which the shaker is connected. The crank is 5 cm. in length and runs at 100 revolutions per minute. If the motor used with the machine is of the series type, it is necessary to have a regulating rheostat, which is usually provided in the base of the motor, to adjust the speed when the shaker is not fully loaded. With a shunt-wound motor, running at practically constant speed, a rheostat is not necessary, providing the reducing gear has been designed to drive the shaker at the proper speed.

This form of shaker is more convenient than one in which the platform is supported from above, since the supports do not interfere with the removal of the trays. The use of thin steel or wooden supports, set with their planes at right angles to the direction of motion, avoids the necessity of guides to prevent lateral motion, while little resistance is encountered to motion in the direction parallel to the plane of the crank. In the apparatus illustrated, the supports are of hickory, 35 mm. wide, 4 mm. thick, and 70 cm. long.

The effect of shaking and the use of ammonia on the mechanical composition of the soil will be discussed later.

THE CENTRIFUGAL MACHINE.

The primary requisite in making mechanical analyses by the centrifugal method is some means of securing the high velocity required to throw down the suspended soil particles. An electric motor is most suitable for this purpose, if an electric-lighting circuit is available. Large direct-current series motors of the desk-fan type are very satisfactory. Alternating-current induction motors of the desk-fan type are not sufficiently powerful to give satisfactory results. In case an alternating-current circuit is the only one available, a belt-driven apparatus used in connection with an induction motor of preferably one-half horsepower capacity should be employed.

The centrifugal apparatus used in this laboratory, and illustrated in Plate II, consists of a 110-volt 16-inch fan motor mounted with its shaft in a vertical position, to which is attached a spider carrying eight trunnioned frames. The distance from the center of the motor shaft to the center of the trunnion screws is 10 cm., and the depth of the trunnioned racks is 15 cm. The centrifugal tubes consist of large heavy glass test tubes 18 by 3 cm., which are supported in the trunnioned racks. The aperture in the upper ring of the support is made large enough to admit the test tube readily, while the opening in the lower ring is smaller than the tube, and is faced with a felt cushion, on which the tube rests. It is important that the tubes should be thoroughly annealed; otherwise breakage is apt to occur under the strain to which they are subjected during rotation. To protect the operator from such accidents a guard surrounds the movable portion of the machine.

The motor is provided with a rheostat in its base, giving four dif-

ferent speeds, which enables one to start the motor slowly and bring it gradually up to full speed. The machine when loaded and running at full speed requires about one minute to stop after the circuit is opened. To avoid this delay the motor is provided with a reversing switch, by means of which the direction of the current through the armature may be reversed and the motor brought quickly to rest. Before stopping the machine in this way the rheostat should be set at the first speed, and then slowly moved to the second or third speed, in order that the motor may not be subjected to too great mechanical and electrical strains in the reversing process.

MICROSCOPE.

A compound microscope is necessary for measuring the sizes of the particles in the silt and clay separations. The instrument should be provided with an eye piece micrometer, and it is of great convenience to adjust the objective and length of the tube so that one scale division of the micrometer will correspond to the superior limit of the smallest group to be measured. In our microscope, using an one-eighth objective, one scale division of the micrometer corresponds to 0.005 mm., which is the superior limit of the clay group. Similarly ten scale divisions, or one large division on the micrometer, corresponds to the superior limit of the silt group. Such an arrangement greatly facilitates rapid measurement.

APPARATUS CONTAINING DISTILLED WATER UNDER PRESSURE.

In washing the samples from the shaker bottles and from the evaporating dishes, and in bringing the soil into suspension after having been packed into the bottom of the tubes by centrifugal action, a jet of distilled water under considerable pressure will be found of great assistance. For this purpose we use a large wash bottle, as shown in figure 1. The air-tight reservoir *R*, having a capacity of 10 liters, is filled by opening the valve *A*, in the pipe leading from the supply tank, the valve *V* being opened also to permit the escape of the compressed air. In case the supply tank is below the level of *R*, the reservoir may still be filled by attaching *R* to a good aspirator pump, providing the distilled water does not have to be lifted more than 25 feet.

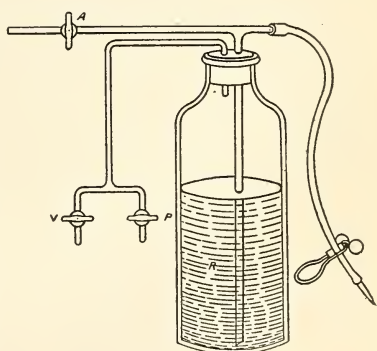


Fig. 1.—Apparatus for obtaining distilled water under pressure.

After filling, *A* and *V* are closed and air is admitted from the pressure cock *P*. If the laboratory is not provided with compressed air,

a large bicycle pump answers the purpose very well. In this case it is important that the connections should be perfectly tight in order to maintain the pressure, and a gauge should be attached to the bottle in order that excessive pressures may be avoided. A pressure of 8 or 10 pounds per square inch gives a jet of sufficient strength to bring the material quickly into suspension after it has been packed firmly in the bottom of the centrifugal tubes. This apparatus is far superior to the ordinary wash bottle, and will well repay the slight trouble incident to its construction.

MAKING SEPARATIONS BY THE CENTRIFUGAL METHOD.

The eight shaker bottles, numbered 1 to 8, are transferred from the shaker tray to a small rack holding them in an upright position. The contents of each bottle are then decanted into the centrifugal tube of corresponding number. It is usually not possible to transfer all of the soil from the shaker bottle to the centrifugal tube in one operation, the remainder of the soil being transferred after one or more decantations of clay water have been made.

Care should be taken in starting the machine to see that the tubes on the opposite sides of the machine contain about the same amount of water, in order that the system may be balanced. It is advisable to arrange the tubes in the machine so that tubes of equal weight shall occupy diametrically opposite positions. The tubes should then be numbered consecutively and this order observed in placing them in the machine.

The tubes being filled, the machine is run at full speed for about three minutes, the time depending upon the speed of the machine and the amount of suspended material present. The machine is then stopped by means of the reversing switch, and the material remaining in suspension examined under the microscope to see if it contains any particles larger than the clay group. A small quantity of suspended material may be conveniently removed for microscopic examination by the use of a small glass tube, about 2 mm. in internal diameter. A finger is placed over the upper end of the tube, and the lower end of the tube inserted to the desired depth in the centrifugal tube. When the finger is removed a small amount of water containing the material in suspension at this depth passes into the tube. The finger is then replaced and the tube withdrawn. A drop of the water containing the suspended material can then be readily transferred to the microscope slide. If only clay particles are found in suspension, the clay water is decanted from each tube into evaporating dishes with corresponding numbers. The remainder of the soil in the shaker bottle is then washed into the centrifugal tubes, the soil in the bottom of the tubes brought into suspension by means of the jet of distilled water, the machine again started, and the separations effected as before. This process should be repeated until all

the particles less than 0.005 mm. in diameter have been removed from the sample.

It is not always possible to remove all the clay without decanting some particles larger than 0.005 mm. in diameter, on account of the irregular shapes of the particles, which modifies the rate of subsidence. This is especially the case with micaceous soils, where the thin flakes or scales do not subside as rapidly as some of the clay particles. In such a case the analyst has but one recourse, namely, to make the loss in one group balance as nearly as possible the gain in the other. This necessitates the direct comparison of the particles which are decanted with those which remain behind in the centrifugal tube. The success of a separation of this kind manifestly depends upon the judgment and experience of the analyst.

The clay suspension is evaporated nearly to dryness on a hot-plate, and is then transferred from the evaporating dishes to small platinum or porcelain dishes for drying and weighing. We have tried a great variety of dishes for evaporating purposes, and the white enameled or granite ware flat-bottom saucepans of about 1 liter capacity seem the most serviceable for this purpose. Porcelain is too fragile and too poor a conductor of heat to be thoroughly satisfactory as an evaporating dish. Aluminum dishes were tried, but they could not be cleaned as thoroughly as the enameled surfaces, and they also reacted with the hot soil solutions, particularly in the case of the alkaline soils. If care is taken to prevent the enameled dish from drying out completely on the drying plate so as to become overheated it will last for a long time. If overheating occurs, however, the enamel soon begins to flake and the dishes are useless for analytical purposes.

The transfer of the clay from the evaporating dish to the platinum dish is easily effected by means of the jet of distilled water, described above, with the help of a small rubber "policeman." The clay suspension in the platinum dish is then evaporated to dryness on a steam bath, dried in the oven at 110° C. and weighed.^a

In the separation of the silts from the sands it is not necessary to use centrifugal force, sedimentation under the action of gravity being sufficiently rapid for this purpose. The centrifugal tubes are accordingly transferred for convenience from the machine to a small rack holding them in a vertical position, and the material brought into suspension by a jet of distilled water as before. Sufficient time is allowed for the coarser particles in the suspended material to subside, a test being made with the use of the microscope before decanting, to be sure that silt only remains in suspension. The material left in the tubes is again brought into suspension, and the operation repeated

^aSmall porcelain dishes glazed on both surfaces may be satisfactorily substituted for the platinum dishes when the separations are not ignited. The porcelain is cleaned rather more easily than platinum and the loss in weight by wear is no greater.

until a test with the microscope shows that no silt remains. The separation of the silt from the sands is the most difficult one in the whole process. The subsidence is so rapid that it is necessary to work quickly, and at the same time great care must be used that some of the particles properly belonging in the sand group are not decanted with the silt.

It is usually not necessary to evaporate the silt water. The silt soon settles to the bottom of the dish, leaving the water above clear or perhaps very slightly turbid. This water can then be decanted and the silt transferred at once to the platinum dish, brought to dryness on the steam bath, dried in the oven, and weighed. The procedure to be followed when the silt water is turbid or contains a considerable amount of soluble material, will be discussed later.

The sands remaining in the centrifugal tubes after the clay and silt have been removed are transferred to the small platinum or porcelain dishes, dried, and weighed, as in the case of the other separations. The sum of the weights of the three separations for each sample should then be equal to 5 grams, and one can thus determine whether loss has occurred during the separation. The sands are then separated into five groups by means of a nest of four sieves. The 1 mm. and 0.5 mm. sieves are of brass with circular perforations. The 0.25 mm. and 0.1 mm. sieves are made of silk bolting cloth stretched over brass frames, and held in position by slip rings. We use for this purpose Nos. 5XX and 13XX of double extra heavy quality silk bolting cloth.

The five separations thus obtained are weighed. The sum of these weights should equal the weight of the sands before sifting, but there is sometimes a slight loss in this process, due to the sand becoming caught in the sieves or lost in transferring to the weighing pan. This loss seldom amounts as much as 10 mg.

Occasionally it will be found that the silt water contains a large amount of soluble material. This condition can be easily shown by measuring the electrical conductivity of the solution. In such cases, if the silt water is decanted and not evaporated, the analysis will foot up short. The correct procedure under these circumstances is somewhat open to question. It appears to us advisable in such cases to class the dissolved material in the silt water with the silt rather than transfer it to the clay. To be sure this dissolved material is neither silt nor clay, but the whole tendency of the analysis of such soils is to make the clay content abnormally high. These soils are found in many cases to consist of small and comparatively insoluble particles cemented into larger aggregates by soluble material. Consequently, when a large amount of water is added, this cementing material dissolves, and the mechanical composition of the soil is greatly changed. This error is to some extent counteracted by the procedure indi-

cated. The mechanical analysis of a soil in which these conditions are observed must be accepted with reserve, since disintegration of the soil must have taken place to a considerable extent during the analysis.

While gypsum soils and soils containing a considerable amount of calcium carbonate are difficult to analyze by the centrifugal method, much better results can be obtained than by those methods in which the soil remains in contact with the water used in the separation for several days. Unless unusual difficulties are met with, the complete separation of the silt and clay in eight samples of soils can usually be made by the centrifugal method in from two to three hours. Occasionally very heavy clay soils will require over three hours. A soil which shows a tendency to disintegrate during analysis will also prolong the time of the analysis, owing to the impossibility of making a sharp separation. As soon as the analyst recognizes this condition it is best to stop without attempting to obtain a sharp separation, as this procedure will more probably give results approximating the true texture of the soil.

LABORATORY RECORD.

The accompanying illustration (fig. 2) represents a page of the mechanical analysis laboratory notebook. The number preceding the word "clay" is the number of the dish in which the separation was dried and weighed. On the first line below is recorded the weight of the dish plus the separation, and on the second line the weight of the dish, the difference giving the weight of the separation; similarly for the "silt" and the "sands." The sum of these three separations should equal the weight of the original sample.

The sands are further divided by the sieves into groups (1) to (5), inclusive, the weight being recorded opposite these numbers. The silt constitutes group (6), and the clay, group (7). The percentages, by weight of the soil particles in each group, are now readily calculated. The final determination of the sum of the weights and of the percentages checks the numerical work.

The conventional names, applied by Whitney to the several separations, are as follows:

- Group (1) Fine gravel.
- Group (2) Coarse sand.
- Group (3) Medium sand.
- Group (4) Fine sand.
- Group (5) Very fine sand.
- Group (6) Silt.
- Group (7) Clay.

For reference purposes, a double record is transcribed from the laboratory book, the results of the analysis being reported to tenths

MECHANICAL ANALYSIS.

Sample No. 10322

Date, Apr 15, 1904

Gravel, > 2 mm., 0 per cent.

ANALYSIS OF 5 GRAMS OF SOIL < 2 MM.

No. 99 Sands (1)-(5).

No. 37 Silt (6).

No. 70 Clay (7).

21.990

23.776

23.028

20.800

21.050

21.945

1.190

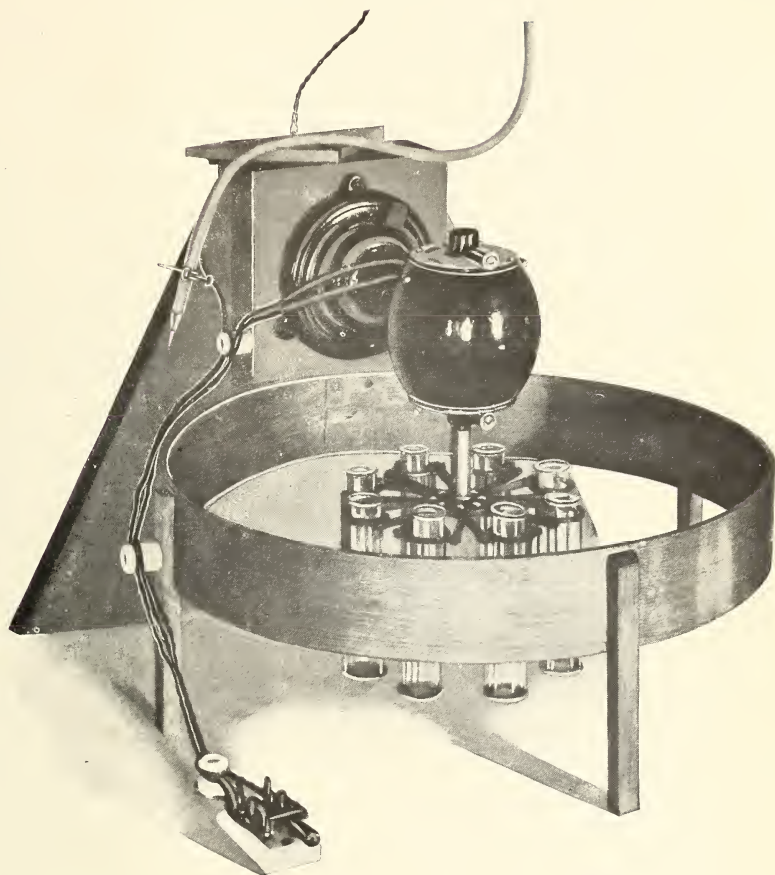
2.726

1.083

	Diameter, mm.		Weight, gms.		Per cent.
(1)	2-1	=	0.008	=	0.16
(2)	1-.5	=	.019	=	.38
(3)	.5-.25	=	.019	=	.38
(4)	.25-.1	=	2.48	=	4.96
(5)	.1-.05	=	.896	=	17.92
(6)	.05-.005	=	2.726	=	54.52
(7)	<.005	=	1.083	=	21.66
TOTAL,			4.999		99.98

Fig. 2.—Laboratory form used in mechanical analysis.

of 1 per cent. One record is made in serial form in a ledger and the other is kept on cards arranged with reference to the locality or area from which the samples were obtained.



CENTRIFUGAL MACHINE USED IN MECHANICAL ANALYSIS OF SOILS.

INVESTIGATION OF VARIOUS FEATURES OF THE CENTRIFUGAL METHOD.

This chapter contains the results of experiments made to determine the effect of certain operations in the centrifugal method of mechanical analysis on the mechanical composition of a soil.

An investigation of the following subjects has been made:

1. The method of subsampling;
2. The effect of oven drying on the mechanical composition;
3. The effect of shaking on the mechanical composition;
4. The effect of the use of ammonia in mechanical analysis; and
5. The accuracy of the centrifugal method.

In the tables in this chapter, the heavy faced numerals in parentheses which head the columns refer to the group number of the mechanical separations (see p. 15), and the numbers immediately below in italics show the limits of the diameters of the particles in each group, in millimeters. The figures in the columns below give the percentage distribution by weight, through the several groups, of the soil grains constituting a given sample. The number of the sample refers to the Bureau catalogue number of the sample.^a

SUBSAMPLING.

Mention has already been made (see p. 9) of the difficulty encountered in securing a representative subsample of a soil containing coarse material, there being always a tendency toward a partial separation of the fine and coarse material in such samples. It has been our practice to mix the samples as thoroughly as possible with a spatula, and then to remove small portions from the various parts of the drying dish to make up the 5-gram sample. This process is necessarily somewhat uncertain, and it occurred to us that the seed-sampling machine devised by E. Brown,^b of the Seed Laboratory of this Department, for obtaining representative subsamples of seed might be used equally well to obtain representative subsamples of soils. This machine has a revolving hopper from which the seed flows through an annular orifice, and is divided into two portions by means of a stationary vertical knife-edge placed just below and bisecting the orifice. In using this apparatus a sample of soil was

^a A catalogue of the first 4,000 samples collected by the Bureau has been published as Bulletin No. 16 of this Bureau. Descriptions of most of the remaining samples may be found in the Reports of the Field Operations of the Bureau of Soils.

^b An illustration and working drawing of this apparatus may be found in Circular No. 34, Revised, Office of Experiment Stations, entitled "Rules and regulations for seed testing." For use in sampling soils the apparatus should be made much smaller, and care should be taken to obtain smooth and polished surfaces in order to prevent the adherence of the finer particles.

placed in the machine and divided into two portions, one of which was again passed through the machine and subdivided. This process was continued until two samples were obtained containing approximately the amount of soil used in an analysis. Samples taken in this manner were analyzed and compared with those taken in the usual way. The results of the analyses are given in the following table:

TABLE I.—*Comparison of methods of subsampling.*

No. of sample.	Weight of sample.	Method of sampling.	(1) 2-1.	(2) 1-0.5.	(3) 0.5-0.25.	(4) 0.25-0.1.	(5) 0.1-0.05.	(6) 0.05-0.005.	(7) 0.005-0.0.
	<i>Grams.</i>		<i>Per cent.</i>	<i>Per cent.</i>	<i>Per cent.</i>	<i>Per cent.</i>	<i>Per cent.</i>	<i>Per cent.</i>	<i>Per cent.</i>
9875 (a)	5.0	Spatula ---	7.2	23.3	8.4	10.9	5.9	25.7	18.2
(b)	5.0	----do----	6.2	22.6	8.9	11.1	6.6	26.8	17.9
(c)	10.5	Machine --	12.8	26.2	7.5	8.3	5.2	23.3	16.6
(d)	9.8	----do----	12.0	25.4	7.7	8.8	5.0	24.3	16.8
9876 (a)	5.0	Spatula ---	8.1	19.4	6.6	7.1	3.9	20.4	34.0
(b)	5.0	----do----	4.2	17.4	7.1	8.3	4.7	24.0	34.1
(c)	7.6	Machine --	12.3	18.0	5.5	5.9	3.4	22.1	32.8
(d)	6.3	----do----	11.3	18.6	5.9	5.6	4.2	22.2	32.2
9881 (a)	5.0	Spatula ---	.4	3.8	10.5	36.4	15.2	22.3	11.4
(b)	5.0	----do----	.5	4.0	11.3	34.0	19.1	21.9	8.4
(c)	5.0	----do----	.5	4.2	11.7	34.3	18.1	22.1	8.6
(d)	5.0	----do----	.4	4.2	10.7	34.9	18.4	22.4	8.4
(e)	5.0	----do----	.3	3.9	11.0	35.3	18.3	21.9	8.4
(f)	5.6	Machine --	.6	5.5	11.8	34.8	18.9	19.9	8.6
(g)	6.3	----do----	.5	4.8	12.1	34.6	19.8	19.7	8.6
(h)	7.4	----do----	.6	4.2	12.3	33.2	20.9	20.3	8.5
(i)	6.7	----do----	.6	4.6	12.3	34.5	20.8	19.2	8.2

Samples 9875 and 9876 were analyzed by F. O. Martin and 9881 by J. R. Pearce. Analyses 9875 (a), 9876 (a), and 9881 (a) were made previous to the other analyses, and are introduced here for comparison.

An inspection of Table I shows that in the case of 9875 and 9876 the percentage of groups (1) and (2) in the samples taken with the spatula is less than when sampled with the machine. These results would indicate that the analyst failed to obtain a sufficient amount of coarse material in subsampling with the spatula. The duplicate analyses of these soils subsampled by the machine also agree better than those in which the samples were taken with the spatula. With 9881 the agreement of the duplicates appears to be about equally good in the two cases. In this sample there is a much smaller percentage of groups (1) and (2), so that the influence of the method of subsampling would not be so evident.

These results show that a mechanical sampler is superior to the spatula for subsampling soils in which groups (1) and (2) are present in any quantity. This applies also to samples in which aggregates exist larger than 0.5 mm. The mechanical sampler has the disadvantage of not giving samples of uniform weight, since the whole of the sample obtained through the last subdivision must, of course, be

used in order to make the method of value. Some inconvenience in calculation is thereby introduced.

Care should be taken to start with a sample the weight of which is approximately 2^x that of the desired sample, x being a positive integral number, and the machine should be so adjusted as to divide the sample into two equal portions. Otherwise the samples obtained may be too large, which prolongs the separation unnecessarily; or too small, which lessens the accuracy of the work. The final subsample should be weighed, which gives a check on the size of the sample and a means of determining whether loss has occurred during the subsequent separations.

EFFECT OF AIR DRYING AND OVEN DRYING ON MECHANICAL COMPOSITION OF SOIL.

It has been our custom to dry soil samples at 110° C. before weighing out the portion for mechanical analysis in order that the work might be checked by the subsequent weighings in the manner already indicated. The question arose whether such oven drying might not modify the mechanical composition of the soil through the formation of aggregates which would not disintegrate in the shaker. For the purpose of investigating this point samples were collected in a moist condition, and after mixing with the hands a portion was first dried at 110° C. and then shaken, while another portion was placed at once in a shaker bottle and water added. The two portions were then analyzed in the usual way. Several determinations were made in this way with samples collected from different points in the Department grounds, the results being given in the following table:

TABLE II.—*Effect of air drying and oven drying on the mechanical composition of soil.*

Number of sample.	Description.	(6) 0.05-0.005.	(7) 0.005-0.
		<i>Per cent.</i>	<i>Per cent.</i>
I(a)	Moist.....	41.3	17.3
(b)	Air dried.....	40.6	17.4
(c)	Oven dried.....	41.7	18.2
II(d)	Moist.....	31.1	26.2
(e)do.....	30.2	26.1
(f)	Oven dried.....	31.7	23.8
(g)do.....	31.5	23.8
III(h)	Moist.....	38.8	26.3
(i)do.....	34.8	27.1
(j)	Oven dried.....	33.5	23.6
(k)do.....	33.6	25.8

Samples (a) to (c), inclusive, analyzed by F. O. Martin; (d) to (k), inclusive, analyzed by J. R. Pearce.

The first three analyses were made without the use of ammonia. In the remainder ammonia was used. The percentage of silt and clay only was determined, since any change in composition due to drying would show most markedly in these separations.

It will be seen from an inspection of the results that in the case of the samples examined oven drying reduces the clay content slightly, with the exception of the first sample, where the clay content was slightly increased. While a definite conclusion can not be drawn without the examination of a large number of samples, the results so far indicate that drying a soil at 110° C. does not seriously modify its mechanical composition, as determined in the moist state. If aggregates were formed to any considerable extent they would be detected in the microscopical examination of the separations during the process of analysis, and such an examination consequently serves to notify the analyst of any tendency in this direction.

EFFECT OF SHAKING ON MECHANICAL COMPOSITION.

In preparing samples for mechanical analysis pestling or shaking in water is necessary to separate the clay from the sand grains. It consequently becomes important to determine the time necessary to effect this disintegration, and whether there is any tendency of the soil to break down through long-continued shaking. The results of a series of experiments made to determine the effect of shaking on the mechanical composition are given in Table III. Nine soils varying widely in mechanical composition were investigated. Five samples of each soil were taken, great care being exercised in taking the sub-samples to obtain uniformity. These samples were shaken for five different periods, varying from one to seventy-seven hours. The shaker used was the one already described in this paper. Ten drops of ammonia were added to each sample before shaking.

To determine more specifically the effect of shaking upon the finer material, the usual clay group in most of the samples was divided into two groups, the limits of which were 0.005 to 0.001 mm. and 0.001 to 0.0 mm.

It will be seen from an inspection of the table that shaking for one hour is in some cases not sufficient to separate the clay from the larger grains. No marked change appears to take place after six hours shaking, except in case of samples shaken for seventy-seven hours, all of which show an increased amount of clay, indicating that in samples shaken for long periods a breaking down of the soil does occur to some extent.

In accordance with the results shown in the table, it has been our practice in preparing samples for mechanical analysis to shake the sample for at least six hours before making the separation.

TABLE III.—*Effect of shaking on mechanical composition.*

No. of sample.	Time in shaker.	(1) 2-1.	(2) 1-0.5.	(3) 0.5-0.25.	(4) 0.25-0.1.	(5) 0.1-0.05.	(6) 0.05-0.005.	(7) 0.005-0.	(8) 0.005-0.001.	(9) 0.001-0.
	Hrs.	P. ct.	Per ct.	Per ct.	Per ct.	Per ct.	Per ct.	Per ct.	Per ct.	Per ct.
303	1	0.0	0.4	0.3	2.1	4.6	43.0	42.6	11.4	31.2
	6	.0	.2	.3	2.3	3.4	39.7	47.1	14.7	32.4
	20	.0	.2	.3	2.4	1.4	43.0	46.0	12.9	33.1
	55	.0	.2	.3	2.8	3.3	40.3	46.7	14.2	32.5
	77	.0	.3	.2	1.9	2.2	40.6	49.3	13.6	35.7
937	1	.3	.6	.5	2.3	3.4	35.1	50.9	6.4	44.5
	6	.4	.7	.3	2.0	4.3	32.4	52.5	7.4	45.1
	20	.5	.6	.3	1.3	1.6	30.5	56.0	6.2	49.8
	55	.2	.9	.4	1.8	2.2	30.9	52.6	7.2	45.4
	77	.4	.5	.3	1.1	1.1	28.1	62.9	6.9	56.0
1328	1	.0	.3	.3	.6	2.5	77.4	14.8	5.5	9.3
	6	.0	.3	.4	.8	3.3	77.3	14.6	7.1	7.5
	20	.0	.3	.3	.6	.3	78.5	16.3	5.7	10.6
	55	.0	.4	.4	.8	.8	79.3	15.4	7.2	8.2
	77	.0	.2	.2	.5	.5	75.2	20.2	6.4	13.8
1343	1	.8	1.8	1.2	1.9	1.5	70.8	19.2	9.2	10.0
	6	1.3	1.8	1.3	1.8	2.3	70.9	17.5	7.5	10.0
	20	1.0	1.5	1.2	1.6	.6	70.3	17.9	7.3	10.6
	55	.8	1.6	1.2	1.6	.6	73.4	17.9	8.7	9.2
	77	1.0	1.5	.9	1.5	.4	66.3	23.8	8.1	15.7
6114	1	1.4	3.6	3.6	10.0	5.0	36.1	30.5	-----	-----
	3	.9	3.5	3.5	8.6	4.8	38.8	28.2	-----	-----
	6	.7	2.9	3.6	9.4	6.2	30.6	37.2	-----	-----
	20	.7	3.2	3.5	9.1	4.6	38.8	30.2	-----	-----
	48	.5	2.9	3.1	8.5	5.6	34.3	35.5	-----	-----
6122	1	4.0	8.8	4.9	13.3	6.6	35.1	22.7	-----	-----
	3	4.7	8.0	4.7	13.0	5.4	35.2	21.5	-----	-----
	6	3.9	8.7	5.0	12.1	8.2	27.8	28.2	-----	-----
	20	5.5	7.1	4.1	12.9	5.0	35.9	24.0	-----	-----
	48	3.0	7.3	5.1	13.2	8.4	31.0	26.9	-----	-----
6146	1	.4	10.2	24.9	38.5	5.6	13.9	5.2	-----	-----
	3	.5	9.8	24.5	38.9	5.0	14.4	6.0	-----	-----
	6	.4	7.9	26.2	39.6	6.9	10.9	7.2	-----	-----
	20	.5	8.7	26.5	39.0	6.3	10.2	7.7	-----	-----
	48	.6	7.6	26.5	39.2	6.8	10.7	7.6	-----	-----
6158	1	.4	.4	.3	.6	.7	38.0	52.8	6.4	46.4
	6	.6	.4	.3	.6	.6	27.8	61.6	16.4	45.2
	20	.3	.5	.3	.6	.3	24.3	66.9	22.4	44.5
	55	.3	.4	.2	.4	.2	22.0	68.8	20.6	48.2
	77	.5	.5	.3	.5	.3	22.0	71.2	17.2	54.0
8035	1	.0	.3	2.2	39.0	24.0	17.0	17.4	-----	-----
	6	.0	.3	2.2	40.3	23.8	16.4	17.0	-----	-----
	20	.0	.4	2.4	37.6	25.1	16.0	18.5	-----	-----
	55	.0	.2	2.1	39.7	24.5	13.4	20.2	-----	-----
	77	.0	.3	2.7	40.9	20.4	15.3	20.3	-----	-----

Analyses made by F. O. Martin.

USE OF AMMONIA IN MECHANICAL ANALYSIS.

Ammonia has a very marked action in breaking up the flocculations which are to be found in most soils containing particles less than 0.005 mm. in diameter. Some investigations have been carried out in this laboratory to determine the effect of the amount of ammonia used on the mechanical composition of the soil. Amounts of ammonia varying from 1 to 60 drops of concentrated ammonia (Baker and Adamson, sp. gr. = 0.9) were added to 5-gram samples of soil, to which 50 cc. of water had been added. These samples were shaken for six hours. Analyses were also made of the same soils shaken without ammonia for the purpose of comparison. The results are given in Table IV.

It will be seen from an inspection of this table that the percentage of clay is greatly increased by the use of ammonia. One drop of ammonia does not seem to be sufficient to break up the floccules completely, but no great change is produced by the addition of more than 5 drops to 50 cc. of water.

TABLE IV.—*Effect of amount of ammonia used on mechanical composition.*

Number of sample.	Quantity of NH ₃ .	(1) 2-1.	(2) 1-0.5.	(3) 0.5-0.25.	(4) 0.25-0.1.	(5) 0.1-0.05.	(6) 0.05-0.005.	(7) 0.005-0.0.
		Drops.	Per cent.	Per cent.	Per cent.	Per cent.	Per cent.	Per cent.
6114	0	0	1.0	3.6	3.4	9.6	5.5	72.7
	1	1	1.3	4.3	3.0	10.1	5.8	44.7
	5	5	.6	4.0	2.8	10.3	6.1	39.6
	10	10	1.4	3.4	3.3	10.4	4.8	40.7
	20	20	1.6	3.5	3.8	9.1	6.1	40.7
	60	60	.9	3.8	2.9	9.7	5.8	40.2
6144	0	0	.0	2.5	5.9	13.7	6.9	56.0
	1	1	.4	2.6	5.2	13.2	4.2	50.4
	5	5	.3	2.3	5.8	13.6	5.4	51.4
	10	10	.1	2.8	5.5	12.9	6.1	51.1
	20	20	.1	2.6	5.8	12.8	6.4	50.5
	60	60	.4	2.3	5.5	13.2	3.9	50.0
6156	0	0	.4	4.1	4.9	4.3	5.4	67.8
	1	1	.5	4.0	5.3	4.8	4.2	62.7
	5	5	.3	4.2	4.9	4.3	4.9	64.3
	10	10	.4	4.3	5.3	5.1	5.2	58.6
	20	20	.7	3.6	4.9	4.3	4.7	63.2
	60	60	.5	4.2	5.2	4.1	4.6	63.1

Analyzed by W. G. Smith and F. O. Martin.

The effect of ammonia is further shown in Table V, in which the analyses of a large number of soils, made both without and with the addition of ammonia, are compared. The first analysis given in the case of each sample was made without ammonia, while in the second analysis 5 drops of ammonia were added to the samples in the shaker bottles. The samples were shaken for six hours in the usual way.

TABLE V.—*Comparison of analyses made without and with ammonia.*

No. of sample	State.	Loss on ignition.	(1) 2-1.	(2) 1-0.5.	(3) 0.5-0.25.	(4) 0.25-0.1.	(5) 0.1-0.05.	(6) 0.05-0.005.	(7) 0.005-0.
		<i>Per ct.</i>	<i>Per ct.</i>	<i>Per ct.</i>	<i>Per ct.</i>	<i>Per ct.</i>	<i>Per ct.</i>	<i>Per ct.</i>	<i>Per ct.</i>
4034	Maryland.....	3.8	3.7	7.6	4.2	4.7	10.9	58.7	6.2
		3.4	4.4	8.0	4.1	4.7	16.1	50.6	8.7
4035do.....	3.0	6.0	12.7	5.6	6.1	8.1	48.7	9.7
		2.6	8.0	14.0	5.4	5.3	12.8	38.3	13.3
*4801	California.....	2.8	.0	.0	.9	2.2	17.2	52.7	22.0
		5.2	.0	.0	2.3	19.5	38.0	26.3	8.7
*4809do.....	5.2	.0	.0	.5	15.3	27.0	24.9	27.8
		3.3	.0	.0	.9	3.6	20.3	57.3	15.0
4817do.....	9.6	.0	.0	1.1	17.1	39.9	24.4	8.0
		7.8	.0	.0	.7	17.4	26.6	34.2	12.8
4967	Pennsylvania.....	5.7	2.5	2.0	2.2	4.4	14.4	49.5	18.8
		6.0	2.8	2.0	2.5	5.0	9.0	47.8	24.4
4968do.....	6.1	.7	1.4	1.5	3.6	21.2	56.8	8.0
		6.9	1.1	1.1	.7	2.9	13.0	61.4	13.9
4969do.....	6.1	.0	.4	.8	3.6	20.1	47.8	21.3
		5.5	.1	1.0	.8	2.7	8.6	47.8	33.5
4970do.....	5.0	1.1	1.8	2.8	4.2	19.2	55.7	10.3
		4.5	1.7	2.3	1.7	4.7	10.6	56.0	18.5
4971do.....	5.9	.7	1.2	1.5	2.9	13.2	43.7	31.5
		5.1	1.9	1.3	.7	1.9	6.3	38.4	44.6
4972do.....	6.8	1.2	2.5	3.3	7.4	29.2	43.3	6.1
		6.0	3.8	2.8	1.9	9.3	22.2	41.9	12.6
4973do.....	3.6	2.3	3.1	4.6	11.3	30.8	37.3	7.1
		2.8	2.0	3.7	2.8	10.9	27.9	34.3	14.5
5021	Ohio.....	7.3	1.0	2.8	7.5	14.0	28.9	30.4	7.2
		6.9	1.4	2.1	2.7	7.3	8.3	54.1	17.1
5022do.....	4.5	.0	1.9	4.3	8.1	28.0	27.4	15.1
		4.9	.7	1.9	1.6	4.9	7.6	52.5	25.9
5023do.....	9.6	2.2	8.5	9.8	11.6	24.1	28.2	5.5
		8.9	.5	2.2	2.4	6.2	9.3	54.4	15.3
5024	Ohio.....	7.3	.0	5.7	8.1	9.7	19.6	38.9	10.4
		5.7	.7	2.3	2.2	6.4	7.5	53.3	22.3
*5027	Utah.....	1.9	.0	1.7	4.5	31.8	17.6	16.2	26.0
		1.6	.0	1.6	4.2	31.9	18.4	17.0	21.9
5116	Maryland.....	2.3	.0	.0	4.5	17.7	23.2	46.0	5.9
		2.2	.0	.0	3.2	17.6	18.8	47.8	9.7
5119do.....	2.7	.0	1.6	2.2	4.6	16.5	57.6	14.6
		2.4	.7	1.2	1.8	4.6	15.5	53.4	20.4
5128do.....	1.9	2.1	4.9	8.1	14.5	10.7	38.4	19.0
		1.6	2.2	4.8	8.5	16.0	10.8	36.4	19.2
5136do.....	2.0	4.3	12.7	13.7	31.2	10.1	10.1	15.2
		1.7	2.9	14.9	14.7	31.0	10.3	8.5	16.2
5137do.....	2.3	.6	3.4	4.2	15.1	18.1	47.9	8.2
		2.2	1.2	4.5	4.6	14.9	13.3	49.9	9.5
5138do.....	2.1	.0	1.0	1.2	28.4	43.7	16.5	5.7
		1.9	.8	4.5	5.8	22.6	10.5	33.8	19.6

TABLE V.—*Comparison of analyses made without and with ammonia—Cont'd.*

No. of sample	State.	Loss on ignition.	(1) 2-1.	(2) 1-0.5.	(3) 0.5-0.25.	(4) 0.25-0.1.	(5) 0.1-0.05.	(6) 0.050.005.	(7) 0.005-0.
		<i>Per ct.</i>	<i>Per ct.</i>	<i>Per ct.</i>	<i>Per ct.</i>	<i>Per ct.</i>	<i>Per ct.</i>	<i>Per ct.</i>	<i>Per ct.</i>
5139	do	2.4	1.1	2.6	4.7	14.7	18.5	47.7	8.3
		2.4	1.0	3.1	4.5	13.4	13.1	49.7	12.8
5159	do	2.8	1.4	7.6	4.6	5.9	13.3	56.8	7.4
		2.6	1.3	7.5	5.9	6.5	14.4	52.0	9.8
5160	do	2.8	2.0	8.3	5.0	4.4	10.8	54.3	12.3
		2.4	1.0	6.2	5.4	5.6	10.3	50.7	18.2
5224	North Carolina	7.6	4.9	8.4	5.8	11.4	3.9	19.4	37.9
		8.9	5.3	8.3	3.7	8.1	3.6	10.9	51.2
*8052	California		.0	.1	.3	22.4	45.1	20.5	11.6
			.0	.1	.3	21.0	42.9	21.8	13.8
*8058	do		.0	.1	.1	14.3	16.5	34.7	34.2
			.0	.1	.1	13.2	17.5	34.6	34.4
*8059	do		.0	.1	.2	23.5	39.8	19.7	16.8
			.0	.1	.1	25.1	38.2	19.6	16.9
*8064	do		.0	.1	.3	59.9	26.0	5.8	7.9
			.0	.0	.5	54.9	31.7	5.1	7.8

Analyses made by George N. Coffey, Charles A. Jensen, and F. O. Martin.

It will be seen from a study of this table that in most samples the effect of the addition of ammonia is to increase the amount of clay. However, in the case of strongly alkaline soils containing more than 1 per cent of soluble material (designated by a star in the table), the addition of ammonia either has little effect or its use has actually diminished the clay content. The latter action is particularly marked in 4801 and 4809. In the case of the last four soils given in the table the samples to which ammonia was added were found to be badly flocculated and the time required for the separation was about twice as great as for the untreated samples.

A consideration of these tables leads to the conclusion that the use of ammonia in mechanical analysis increases the percentage of group (7) in soils that are not alkaline by destroying the formation of flocules or aggregations of the smaller particles. In the case of strongly alkaline soils the addition of ammonia appears to flocculate the soil rather than break up the flocculations, and its use in such cases is therefore not desirable. From 5 to 10 drops of ammonia added to 5 grams of soil in 50 cc. of water appears to be sufficient to break up the flocculations in nonalkaline soils.

In case a soil contains a considerable amount of lime or magnesium carbonate flocculation will occur, which usually becomes more pronounced upon the addition of ammonia. This action was well illustrated in two samples of Houston clay (10324 and 10325) from the Macon area, Georgia. These were badly flocculated at the beginning of the mechanical analysis, but after two or three decantations had

been made the tendency to flocculate disappeared and the final separation was made without difficulty. Subsequent chemical analyses of these samples showed them to contain 25.8 per cent and 42.1 per cent, respectively, of carbonates calculated as calcium carbonate.

ACCURACY OF MECHANICAL ANALYSIS BY THE CENTRIFUGAL METHOD.

The following table gives the results of the analyses of eight 5-gram samples of the same soil, the analyses of all the samples being made at the same time. The subsamples were taken with the spatula, the regular laboratory procedure of mixing and quartering being followed. These analyses may fairly be taken to represent the limit of accuracy attainable by the centrifugal method under the most favorable conditions.

TABLE VI.—*Accuracy of mechanical analysis as shown by the analyses of eight 5-gram samples of the same soil.*

No. of sample.	Loss on ignition.	(1) 2-1.	(2) 1-0.5.	(3) 0.5-0.25.	(4) 0.25-0.1.	(5) 0.1-0.05.	(6) 0.05-0.005.	(7) 0.005-0.
	<i>Per cent.</i>	<i>Per cent.</i>	<i>Per cent.</i>	<i>Per cent.</i>	<i>Per cent.</i>	<i>Per cent.</i>	<i>Per cent.</i>	<i>Per cent.</i>
5862(a) -----	12.5	2.6	5.3	4.2	13.0	7.7	16.8	38.3
(b) -----	12.2	2.6	5.1	4.8	13.2	7.3	17.6	37.5
(c) -----	10.9	2.6	5.1	4.6	14.2	8.8	15.8	38.1
(d) -----	12.2	2.4	5.1	4.5	13.8	7.2	16.7	37.9
(e) -----	12.0	2.4	5.3	4.4	14.4	7.8	16.0	38.0
(f) -----	12.2	2.3	5.6	4.5	12.8	8.0	16.4	38.0
(g) -----	12.4	2.8	5.6	4.9	13.6	8.4	15.1	37.6
(h) -----	11.9	3.2	5.2	4.4	12.6	7.3	17.2	37.7

Analyses made by J. O. Martin.

OTHER METHODS OF MECHANICAL SOIL ANALYSIS.

HILGARD'S ELUTRIATOR METHOD.

Professor Hilgard was the first in this country to appreciate the importance of mechanical analysis, his first paper^a on the subject appearing in 1873. Hilgard's method depends upon the fact that a stream of water moving vertically upward through a tube will suspend and carry off soil particles below a certain diameter, depending upon the velocity of the water. By thus regulating carefully the velocity of the stream one can effect the various separations desired. Hilgard's apparatus, which he terms the "churn elutriator," resembles Schöne's apparatus, but includes two important modifications, (1) the substitution of a cylindrical tube for the vertical conical tube used by Schöne, which eliminates the eddies formed in a conical tube, and thus secures greater uniformity in the sizes of the particles removed; (2) the addition of a rapidly revolving stirrer at the lower end of the

^a Am. Jour. of Science, series 3, Vol. VI, 1873, pp. 288-296, 333-339.

elutriator tube to break up flocculent masses. The general arrangement of the apparatus and connections is shown diagrammatically in figure 3.^a

The stirrer should be run at a speed from 500 to 600 revolutions per minute in order to be effective. The agitation of the water by the stirrer is prevented from being communicated to that portion in which the separation is made by means of a wire screen of 0.8 mm. aperture cemented across the bottom of the cylinder. To secure con-

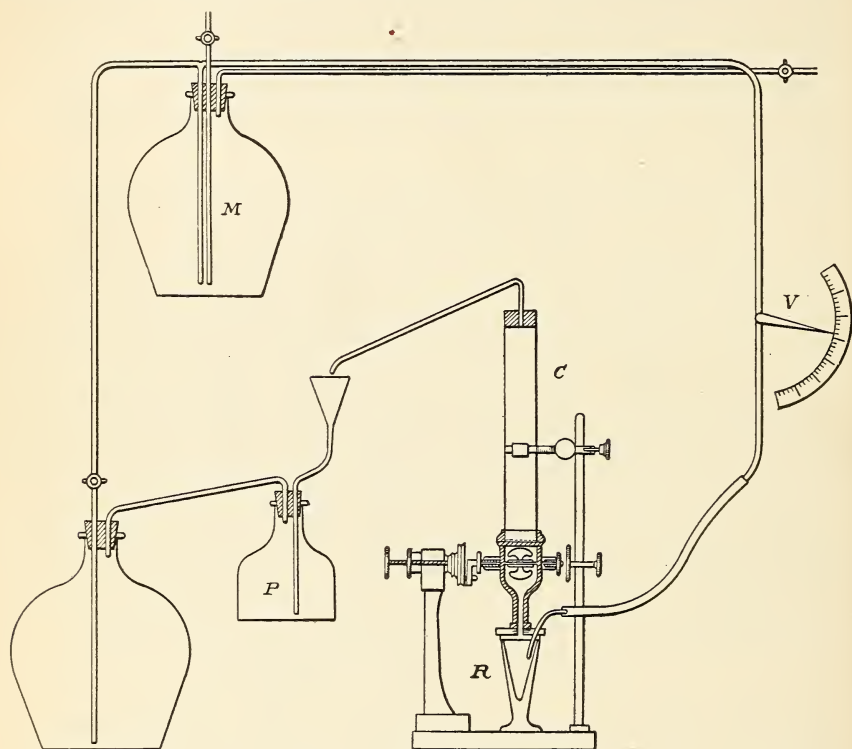


Fig. 3.—Hilgard's elutriator.

cordant results it is necessary that the tube of the instrument should be as nearly cylindrical as possible and placed in a vertical position. Sunshine or the proximity of other sources of heat must be avoided. The amount of sediment discharged at any one time must not exceed that producing a moderate turbidity, and clear water must be used. Distilled water is not necessary.

Hilgard uses the elutriator only in separating particles larger than 0.01 mm. in diameter, this material being first separated from the smaller particles by subsidence. The material to be analyzed by the elutriator is introduced into the reservoir *R*. A constant pressure of

^a For a full description of the method, see Rep. of Cal. Exp. Sta., 1891-92, p. 243.

water is secured by means of a Mariotte apparatus, M , the velocity of the stream being controlled by the graduated valve V . Each separation is, of course, associated with a considerable volume of water; however, by allowing the water coming from the cylinder C to flow into a vessel of large horizontal cross section P , the velocity of the stream will be much lessened, and the suspended material will in consequence sink to the bottom of the vessel. In this way the separation can be confined to a comparatively small volume of water.

Hilgard expresses his various separations in terms of their "hydraulic value;" that is, the upward velocity of the water column in millimeters per second, which is just sufficient to remove the separation.

The following table, taken from Hilgard's paper, shows the various hydraulic values he employed, with the corresponding diameter of the quartz grains removed at these velocities:

TABLE VII.—*Diameters and hydraulic values of sediments.*

Diameter of quartz grains.	Velocity per second, or hydraulic value.	Diameter of quartz grains.	Velocity per second, or hydraulic value.
<i>mm.</i>	<i>mm.</i>	<i>mm.</i>	<i>mm.</i>
0.50	64	0.047	2
0.30	32	0.036	1
0.16	16	0.025	0.5
0.12	8	0.016	0.25
0.072	4	0.010	< 0.25

Material less than 0.25 mm. hydraulic value Hilgard separates into two portions by subsidence. The material remaining in suspension after subsidence for twenty-four hours in a column of water 20 cm. in height he terms "clay." The remainder of the material is termed "fine silt." This clay is precipitated by adding 50 cc. of saturated solution of sodium chloride to each liter of clay water, the precipitation being accelerated by warming. The precipitated clay is collected in a filter, washed with weak brine, dried at 100° C., and weighed. It is again placed in the funnel, washed with a weak solution of ammonium chloride until the sodium chloride is removed, the filtrate evaporated, and the residue ignited and weighed. Its weight deducted from the total weight gives the amount of clay.

OSBORNE'S BEAKER METHOD.

Osborne^a in using the method of Moore,^b in which the soil particles were allowed to settle in a cylindrical column of water for a given interval of time, found that the separations could be made more simply and easily by systematically repeated decantations from beakers,

^a Annual Report, Conn. Agr. Expt. Sta., 1886, p. 141; 1887, p. 144; 1888, p. 154.

^b Tenth U. S. Census, vol. 3, pp. 872-873.

the results being controlled by microscopic measurements. Osborne expresses the fundamental principles of his method as follows:

Make the duration of the subsidences such that the liquid decanted the first few times shall contain nothing *larger* than the desired diameter. Then decant into another vessel, timing the subsidence so that the sediment shall contain nothing *smaller* than the chosen diameter. This can not be done without decanting much that is larger than the chosen diameter, but the greater part of the particles greater and less than the chosen diameter can be removed and an intermediate product obtained, the diameters of whose particles are not very far from that desired.

The reason for first removing the greater part of the coarse particles lies in the fact that as they rapidly subside they drag down with them much of the fine material and render it difficult to effect a sharp separation in their presence. If, then, we bear in mind the general principle that repeated subsidences and decantations properly timed will gradually remove the fine particles from the coarse, and also the fact that the removal of the particles much above and much below the limits of any desired grade greatly facilitates the separation of that grade, there will be no difficulty in obtaining satisfactory results after a little practice.

Osborne ^a recommends ammonium nitrate as a precipitant in determining the clay. By bringing the clay to dryness in a platinum dish and treating several times with hot water the salt can be largely removed before ignition. The clay remains in the bottom of the dish and the salt solution can be drawn off with a pipette. This avoids the snapping which otherwise is liable to occur during ignition. Ammonium nitrate is preferred to ammonium chloride, as there is no danger of loss of iron due to the formation of the volatile ferric chloride.

The Osborne method was employed in the earlier work of the Division of Soils.^b The clay was not precipitated, the determination being made by evaporating an aliquot part of the clay water. Flocculation was prevented either by the addition of a few drops of ammonia or by vigorously stirring the solution with an egg beater.

KING'S ASPIRATOR METHOD.

King ^c states that the results of mechanical soil analysis, as ordinarily expressed, do not furnish a basis for determining either the water capacity of the soil or the rate at which water and air may move through the soil. He has accordingly formulated a method for determining the "effective diameter" of the soil particles. In this method is taken into consideration the grouping of the soil grains, upon which the rate of percolation and water-holding capacity of the soil depends. The procedure consists in determining the rate at which air passes through a column of air-dried soil of a given cross section and

^a Annual Report, Conn. Sta., 1898, p. 156.

^b Bulletin No. 4, Division of Soils, 1896.

^c Fifteenth Annual Report, Wisconsin Expt. Sta., 1897, p. 123; Nineteenth Annual Report, U. S. Geological Survey, Pt. II, 1897-98, pp. 59-294.

height under standard conditions of temperature and pressure. The apparatus used is shown diagrammatically in figure 4. *A* is the aspirator from which an air tube leads to the chamber *C*. The soil tube *D* having a capacity of 100 or 200 cc. and provided with a wire gauze

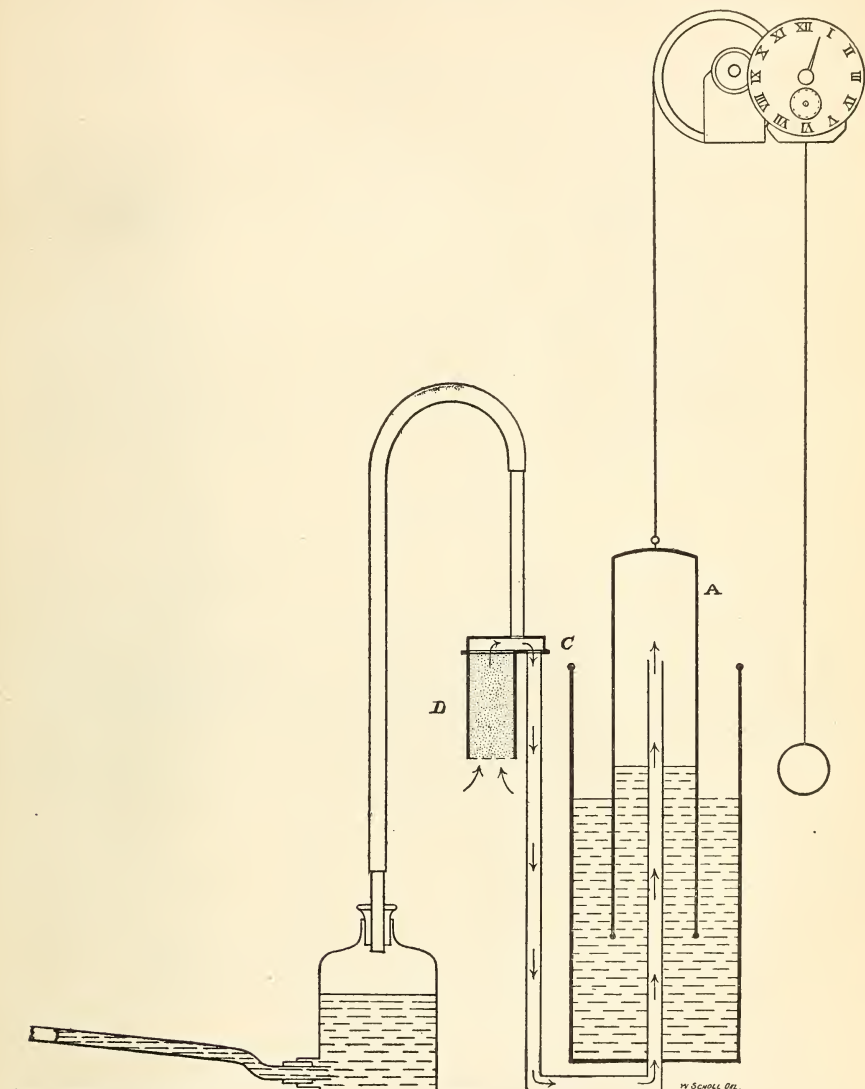


Fig. 4.—King's aspirator for determining the "effective diameter" of soil particles.

bottom, is filled with air-dried soil and connected to *C*. The cord lifting the aspirator bell passes over a pulley to which an air meter is geared, a suitable weight being attached to the other end of the cord. An inclined water pressure gauge capable of being read to tenths of a millimeter is also connected to the air chamber.

The preliminary preparation of the soil, if the object of the determination is to obtain the effect of the soil in its most finely divided condition, consists in pestling the air-dried sample in a mortar with a rubber-tipped pestle. Another preliminary treatment is given when the investigation of the "normal crumb structure" of the soil is desired.

In filling the tube, a solid cap is screwed on in place of the wire gauze. The sample is introduced in small lots of from 3 to 5 cc. and tapped with a flat-faced pestle until the tube is level full. The tube is then held rigidly against the table and jarred by repeated light blows, as long as any settling can be produced, the tube being kept full as the settling progresses. The object is to secure the closest possible packing. Violent jarring or failure to press the tube firmly on the table while packing will loosen the soil.

King expresses his results in terms of the "effective diameter" of the soil grains; that is, the diameter of the grains in a soil composed of spherical particles of uniform size, which, when arranged in the most compact manner possible, have the same permeability as the soil under investigation. In obtaining this diameter, the following formula, due to Slichter,^a is used:

$$d^2 = .878 \frac{hk}{sp t}$$

where

d =diameter of grain in cm.

h =length of sand column in cm.

s =area of cross section of sand column in sq. cm.

p =pressure in cm. of water at 20° C.

t =time in sec. for 5,000 cc. of air to flow through at a temperature of 20° C.

The value of k is determined from the following table, being dependent upon the pore space, which must be measured independently.

Per cent of pore space.	Log. k.	Per cent of pore space.	Log. k.
26	1,9258	37	1,4173
27	1,8695	38	1,3816
28	1,8195	39	1,3445
29	1,7701	40	1,3078
30	1,7199	41	1,2725
31	1,6732	42	1,2374
32	1,6277	43	1,2024
33	1,5847	44	1,1690
34	1,5409	45	1,1370
35	1,4999	46	1,1058
36	1,4592	47	1,0729

YODER'S CENTRIFUGAL ELUTRIATOR METHOD.

Yoder^b has recently combined the fundamental principles of the centrifugal and elutriator methods in an apparatus which he calls the

^aNineteenth Annual Report U. S. Geological Survey, Pt. II, 1897-1898, pp. 301-384; Water Supply and Irrigation Papers, No. 67, U. S. Geological Survey, 1902.

^bBulletin No. 89 of the Utah Exp. Sta., 1904.

“centrifugal elutriator.” The method is essentially an elutriator method, in which the particles are acted upon by a force much greater than that due to gravitation; consequently in removing particles of any given size a velocity of the water column may be employed much larger than would be possible in an elutriator method operating under the action of gravity.

The centrifugal machine used by Yoder is shown with the elutriator

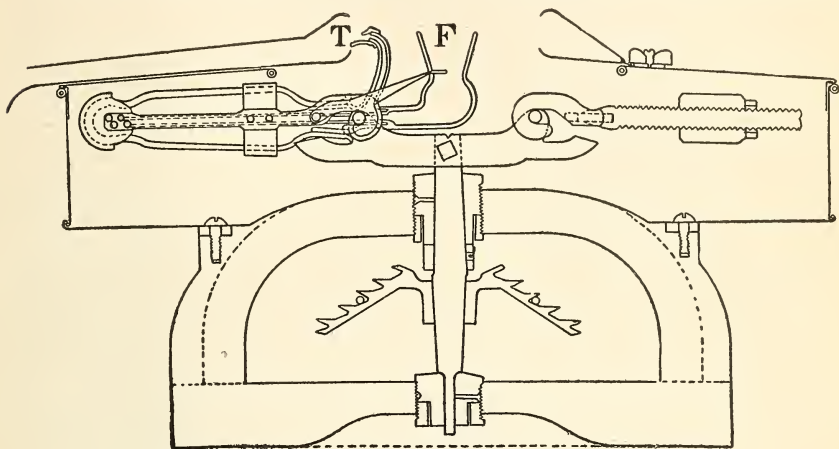


Fig. 5.—Yoder's centrifugal elutriator; vertical section.

bottle in position in figure 5. In figure 6 a longitudinal section of the elutriator bottle is shown. This bottle is so designed that in the portion in which the separation is effected the velocity of the water column and the centrifugal force decrease in the same ratio, as the axis of the machine is approached. A suspended particle is consequently subjected to a uniform force while in this portion of the bottle.

It was not found practicable to stir the sediment in the bottom of

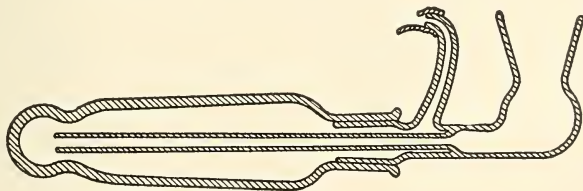


Fig. 6.—Centrifugal elutriator bottle; longitudinal section.

the bottle by the inflowing current of water, consequently the material to be separated is gradually introduced into the machine suspended in a stream of water. The centrifugal elutriator is used only in separating particles less than 0.03 mm. in diameter, this finer material being first separated from the rest of the sample by a gravity elutriator. The water containing this fine material in suspension is run at a uniform rate through the machine, being introduced into the

bottle through the funnel *F*, the axis of which coincides with the axis of the centrifugal machine, and coming out at the opening *T*, where it is caught by the spray collector shown in the upper part of figure 6. The size of the particles which pass out of the machine suspended in the water depends both upon the velocity of the stream of water and upon the speed of the machine.

A uniform flow of water containing the suspended material is secured in the following manner: The water containing the suspended material is placed in a funnel closed at the top, from which it flows into the machine as fast as air is admitted into the funnel. The air comes from a large closed bottle serving as an air chamber, into which the water flows from a Mariotte apparatus, placed above, through a graduated valve that can be regulated at will.

The machine is first operated at such a speed that all particles larger than the superior limit of the group containing the finest material are precipitated to the bottom of the elutriator bottle. During this process some of the finest material is also carried down with some of the largest particles. The machine is then stopped, the material in the bottom of the centrifugal bottle again suspended in water, and the operation repeated with the machine running at such a speed that the soil grains belonging to the group of coarsest particles will alone be precipitated. This process is continued until a sharp separation is obtained. The same procedure is followed for the other separations. The material remaining in suspension at the end of the final separation is precipitated by the addition of a solution of sodium chloride, the amount of salt in the sediment being determined volumetrically by titration with silver nitrate.

VARIOUS SYSTEMS OF GROUPING EMPLOYED IN MECHANICAL ANALYSIS.

Several different systems of grouping the soil particles in a mechanical analysis have been adopted in the United States. Professor Whitney,^a in the early work of the Division of Soils, adopted the grouping of Osborne, but extended it by dividing several of the groups into two parts. This plan has been followed in the later work of the Bureau, with the exception that the two silt groups have been combined, as the advantage of this division in soil classification did not subsequently appear to be sufficient to justify the additional work required for the separation. Hopkins^b has proposed a system of grouping in which, with the exception of the clay group, the superior limit of each group is $\sqrt{10}$ times that of the inferior limit. Hilgard's grouping with the elutriator has already been given. The four sys-

^a Bulletin No. 4, Division of Soils, U. S. Dept. of Agr., 1896.

^b Proceedings Fifteenth Annual Convention, Association of Official Agricultural Chemists, Bulletin No. 56, Division of Chemistry, U. S. Dept. of Agr., 1897, p. 64.

tems which have been used most extensively in the United States, namely, the Hilgard, the Osborne, the Bureau of Soils, and the Hopkins systems, are given below in tabulated form for the purpose of comparison:

TABLE VIII.—*Systems of grouping employed in mechanical analysis.*

Number of group.	Hilgard.	Osborne.	Bureau of Soils.	Hopkins.
	<i>mm.</i>	<i>mm.</i>	<i>mm.</i>	<i>mm.</i>
(1)	3.0	3.0	2.0	1.0
(2)	1.0	1.0	1.0	.32
(3)	.5	.5	.5	.1
(4)	.3	.25	.25	.032
(5)	.16	.05	.1	.01
(6)	.12	.01	.05	.0032
(7)	.072	(?)	.005	.001
(8)	.047	-----	-----	-----
(9)	.036	-----	-----	-----
(10)	.025	-----	-----	-----
(11)	.016	-----	-----	-----
(12)	.010	-----	-----	-----
(13)	(?)	-----	-----	-----

The numbers given in the table represent the diameters of the particles forming the limits of adjacent groups. Hilgard's system contains thirteen groups, while the other systems have seven. In our opinion Hilgard carries his subdivision rather too far to make the system a practical one for soil classification. Even seven groups become rather cumbersome in comparing the composition of a number of samples. It will be noted that Hopkins separates the material below 0.1 mm. diameter into five groups, while the Bureau of Soils divides this material into three groups. The greater number of divisions in the finer material greatly increases the labor of a mechanical analysis, and the separation of particles as small as 0.001 mm. is attended by considerable uncertainty. We have concluded from a determination of the amount of clay less than 0.001 mm. in diameter in a number of soils that the advantages to be gained by such a division do not in general compensate for the additional labor involved. Occasionally the more detailed analysis is made of a sample which exhibits peculiar properties. The advisability of dividing the clay group may, however, fairly be considered an open question, and should be made a subject of further investigation.

DETERMINATION OF ORGANIC MATTER.

Certain physical properties of soils are influenced by the amount of organic matter present, and a method for its accurate determination is consequently of importance.

In the earlier work of the Bureau the amount of organic matter in

a soil was determined from the loss on ignition. This method gave erroneous results in the case of many soils, water also being driven off at the high temperatures necessary to effect the complete combustion of the organic matter. The ignition method has consequently been superseded by the chromic-acid method, which is not influenced by the amount of combined water in the soil. The following description of this method is taken from a paper by Cameron and Brezeale,^a of the chemical laboratory of this Bureau, to whom we are also indebted for a description of some modifications in the procedure made since their paper was published.

THE CHROMIC-ACID METHOD OF DETERMINING ORGANIC MATTER.

The combustion is effected in a round-bottomed flask *F*, figure 7, of about 400 cc. capacity, fitted with a three-hole rubber stopper.

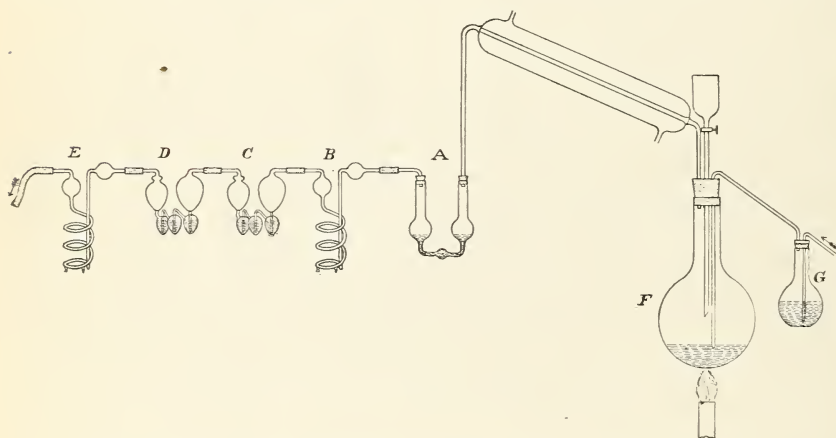


Fig. 7.—Apparatus for determining organic matter by chromic-acid method.

The stopper is fitted with a dropping funnel, a tube for the introduction of air previously freed from carbon dioxide by bubbling through a solution of potassium hydrate in the flask *G*, and a tube leading through a condenser to a train of absorption bulbs. This train contains, first, a Peligot tube *A*, containing a saturated and slightly acidified solution of silver sulphate to absorb both hydrochloric acid and sulphur trioxide or dioxide should they be generated; then a guard tube *B*, containing concentrated sulphuric acid, followed by a potash bulb *C*, and an acid bulb *D*, to be weighed with the potash bulb. An acid guard bulb *E* completes the train. The whole apparatus is attached to an aspirator, so that air free from carbon dioxide can be drawn through the combustion flask and train. The procedure is as follows:

“A sample of the soil, usually about 10 grams, is carefully weighed

^aJourn. Am. Chem. Soc., 26, 29, 1903.

and brought into the combustion flask. If the sample be rich in organic matter, it has been found advisable to introduce also some sand, previously ignited before the blast, and in an amount dependent roughly upon the apparent quantity of organic matter in the soil. From 5 to 10 grams of pulverized potassium bichromate are then added, and the whole mixed thoroughly by shaking, care being taken to prevent any of the mixture adhering to the sides of the flask above the level of the mixture. The flask is closed securely by the stopper, and a gentle stream of air drawn through the whole apparatus by means of the aspirator. When this stream of air has been passing for about ten minutes, concentrated sulphuric acid (sp. gr. about 1.83) is slowly and cautiously run in by means of the dropping funnel until the tip of the glass tube for the introduction of air is covered. When this point has been reached, and if no very vigorous action is taking place, the combination flask is slowly heated until the sulphuric acid commences to give off fumes. It is held at this temperature for from five to ten minutes, and then allowed to cool slowly, unless there is reason to believe combustion has not been complete, in which case the temperature is again raised. Care must be exercised to see that a steady current of air be kept passing through the apparatus, and that the mixture in the flask be not forced back toward the wash bottles. If necessary, quite a rapid stream can be drawn through the absorption bulbs without much risk of losing the determination. It is advisable to have the bulb of the dropping funnel empty before commencing the heating, so that the tube can be quickly opened. In over 400 experiments with this method the flask broke but once, and then the dropping funnel could not be opened because it contained a quantity of sulphuric acid. A sudden large increase of pressure was generated in the flask, owing to faulty manipulation. The dangerous character of such an accident is sufficiently obvious, but with ordinary care liability of its occurrence is extremely small."

MODIFICATIONS FOR SOILS CONTAINING CHLORIDES AND CARBONATES.

"In many soils from arid, semiarid, or marshy areas, there is a considerable content of chlorides. By following the procedure just described with these soils, chlorine gas may be generated, which would be collected in the potash bulbs, forming a mixture of the chloride and hypochlorite in proportions difficult to estimate accurately, and vitiating any attempt to determine the amounts of carbon dioxide absorbed. We have made a number of attempts to get around this difficulty, and have found that it can be met quite simply. If the bichromate of potash be not mixed with the sample before running in the concentrated sulphuric acid, but be dissolved in the acid itself and the solution be slowly and cautiously run in upon the soil, with no attempt to heat the mixture until the reaction in the flask has pro-

ceeded for some time, no hydrochloric acid, chlorine, nor chromyl chloride gas is generated, or in but very small amounts. The procedure thus modified has been used a large number of times with artificial mixtures and natural soils, and has proved satisfactory, although no explanation is obvious why hydrochloric acid should not be formed and oxidized under these conditions. We can only say that, although we discovered the fact empirically, we have thoroughly tested it with the most gratifying results for the method.

“When the amount of chlorides is relatively large, it has sometimes been found desirable to treat the sample with a small volume of dilute sulphuric acid, adding more acid in small quantities from time to time, if necessary, to digest on the steam bath until the major part of the hydrochloric acid has been removed, and to evaporate as much of the water remaining as can be done without permitting a noticeable action of the solution upon the organic matter. The combustion is then carried out as above described.

“With soils which contain carbonates of the alkalies or alkaline earths, it would probably be found satisfactory first to treat with sulphurous acid to decompose the carbonates, and drive out the carbon dioxide, without oxidizing the organic matter and then to get rid of the water and sulphurous acid by evaporating to dryness before proceeding with combustion.

“This method presents, however, a number of difficult manipulations, and requires a great deal of time. It has been found, in the experience of this laboratory, much more convenient to make a separate determination of the carbon dioxide liberated from carbonates, by treating a separate sample of the soil with dilute sulphuric acid (1:6 by volume), and subtracting the amount thus found from the total obtained in the combustion. While this method is not entirely free from objections for very accurate work, it does unquestionably lead to values with all the accuracy necessary for most purposes to which the determination of the organic matter in a soil is applicable.”

COMPARISON OF RESULTS OF VARIOUS METHODS OF DETERMINING ORGANIC MATTER.

Cameron and Breazeale have made a comparison of various methods used in determining organic matter, the results of which are given in Table IX.

TABLE IX.—*Comparison of organic matter, humus, and loss on ignition.*

Sample No.	Loss on ignition.	Humus, official method.	Humus, using pump.	Combustion, in tube, $\text{CO}_2 \times 0.471$.	Combustion, wet way, $\text{CO}_2 \times 0.471$.
	<i>Per cent.</i>	<i>Per cent.</i>	<i>Per cent.</i>	<i>Per cent.</i>	<i>Per cent.</i>
4248	3.92	1.27	0.47	0.87	0.67
4249	6.67	1.40	.70	.97	.74
5223	3.58	1.30	.70	2.26	1.75
5224	7.63	3.45	.30	1.47	.68
5861	6.76	2.05	.90	2.54	2.18
5862	11.98	2.80	.15	.98	.38

Six samples of soils were investigated, the first, third, and fifth samples being surface soils, and the remaining samples the corresponding subsoils.

The results given in the several columns of Table IX were obtained as follows: The soils in column 2 were brought to a red heat in a muffle furnace, after which the samples were moistened, treated with carbon dioxide under pressure, dried at 110° , and weighed; in column 3 the official method of the Association of Official Agricultural Chemists was used; in column 4 the ammoniacal solution obtained by the official method was first filtered through the Chamberland-Pasteur filter tube to remove the suspended clay; in column 5 the amount of CO_2 formed in burning the soil in a tube in a combustion furnace was determined; in column 6 the chromic acid method described above was employed.

It will be noted that the chromic acid method gives results which agree fairly well with those obtained by combustion in a tube, although the dry combustion method gives higher results in every case. This difference is ascribed by Cameron and Breazeale to carbon dioxide held in the soil either in chemical combination or occluded mechanically.

In determining the percentage of organic matter in the soil from the percentage of carbon dioxide found it is of course necessary to use a conversion factor, which, multiplied by the weight of CO_2 , gives the weight of organic matter. The factor generally used for this purpose is 0.471, based upon Wollny's investigation of the percentage of carbon in the humus of the soil. This subject was further investigated by Cameron and Breazeale, who found from an examination of 19 soils that the percentage of carbon in the organic matter present averaged about 42 per cent, instead of 56 per cent, the figure accepted by Wollny, Wolff, van Bemmelen, and others. Wiley^a states that this percentage may vary from 42 to 72 per cent. While Cameron and Breazeale's determinations vary from 33 to 50 per cent, they have

^a Wiley's *Agricultural Analysis*, vol. 1, contains a very complete historical account of this subject.

recommended that the factor 0.471 be retained, with the understanding that it is a conventional factor, and that consequently a determination of the organic matter in a soil by its use must be regarded as only an approximation to the truth by a purely conventional method.

The amount of organic matter in a soil may quickly be calculated from the weight of carbon dioxide liberated by the use of the following table, for which we are indebted to J. G. Smith, of the chemical laboratory of this Bureau. The table is calculated on the basis of the van Bemmelen factor (0.471) and on the supposition that a 5-gram sample of soil is used in the determination. If 10 grams of soil are used, divide the percentage in the table by 2. For a 1-gram sample, multiply the percentage by 5:

Table of percentages of organic matter corresponding to weights of carbon dioxide in 5 grams of soil.

Weight CO ₂ .	O. M.	Weight CO ₂ .	O. M.	Weight CO ₂ .	O. M.	Weight CO ₂ .	O. M.	Weight CO ₂ .	O. M.
<i>Gram.</i>	<i>Per ct.</i>	<i>Gram.</i>	<i>Per ct.</i>	<i>Gram.</i>	<i>Per ct.</i>	<i>Gram.</i>	<i>Per ct.</i>	<i>Gram.</i>	<i>Per ct.</i>
0.0101	0.1	0.1163	1.1	0.2224	2.1	0.3307	3.1	0.4348	4.1
.0207	.2	.1269	1.2	.2331	2.2	.3362	3.2	.4454	4.2
.0314	.3	.1375	1.3	.2437	2.3	.3498	3.3	.4560	4.3
.0420	.4	.1481	1.4	.2543	2.4	.3605	3.4	.4666	4.4
.0526	.5	.1588	1.5	.2649	2.5	.3711	3.5	.4772	4.5
.0632	.6	.1694	1.6	.2755	2.6	.3817	3.6	.4878	4.6
.0738	.7	.1800	1.7	.2861	2.7	.3923	3.7	.4985	4.7
.0844	.8	.1906	1.8	.2968	2.8	.4029	3.8	.5091	4.8
.0951	.9	.2012	1.9	.3074	2.9	.4135	3.9	.5197	4.9
.1057	1.0	.2118	2.0	.3180	3.0	.4241	4.0	.5303	5.0

